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IS 14128 (1994): N-(1,3-Dimethylbutyl)-N~-Phenyl paraphenylenediamine (6 PPD) [PCD 13: Rubber and Rubber Products]

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एन-(1, 3-डाइमिथाइलब्यूटाइल)-एन´-फिनाइल पेराफिनाइलीनडाइएमीन (6 पीपीडी) – विशिष्टि

Indian Standard

N-(1, 3-DIMETHYLBUTYL)-N′-PHENYL PARAPHENYLENEDIAMINE (6 PPD) — SPECIFICATION

UDC 678·6·523

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1994

Price Group 2

Rubber Products Sectional Committee, PCD 13

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

N-(1, 3-Dimethylbutyl)-N′-phenyl paraphenylenediamine (6 PPD) is the most widely used antidegradant of the paraphenylenediamine class. It offers excellent protection to all types of general purpose elastomers against heat, oxidation, ozone, flex-fatigue and metal ion catalysed oxidation without getting extracted in water. It is used at 1·0 — 4·0 phr levels along with blended waxes in rubber compounds intended for use in heavy and light duty truck tyres, passenger tyres, tyre sidewalls, retreading materials, conveyor belts and other industrial products requiring high performance. It is a staining type antidegradant and does not bloom at the levels indicated above. It is soluble in ethyl alcohol and acetone, moderately soluble in hydrocarbon solvents and insoluble in water. Its typical specific gravity is 0·995 at 60°C/15°C. For general purpose applications it is often used in combinations with other general purpose antioxidants for achieving higher product performance.

This standard describes chemical requirements and recommended procedure for the evaluation of N-(1, 3-Dimethylbutyl)-N′-phenyl paraphenylenediamine (6 PPD) in a standard natural rubber based test compound.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

N-(1, 3-DIMETHYLBUTYL)-N′-PHENYL PARAPHENYLENEDIAMINE (6 PPD) — SPECIFICATION

1 SCOPE

This standard prescribes the requirements, methods of sampling and test for N-(1, 3-dimethylbutyl)-N′-phenyl paraphenylenediamine (6 PPD) intended for use as an antidegradant in rubber compounds.

2 NORMATIVE REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revisions and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1675 : 1971	Stearic acid, technical (*first revision*)
3399 : 1993	Zinc oxide for rubber industry (*second revision*)
3660 (Part 51) : 1972	Method of test for natural rubber : Part 51 Determination of ash, total copper, manganese, rubber hydrocarbon, viscosity (shearing disc viscometer) and mixing and vulcanizing of rubber in standard compound (*first revision*)
4588 : 1986	Rubber, raw, natural (*third revision*)
6918 : 1972	Mercaptobenzothiazole
7069 : 1986	Benzothiazyl-2-cyclo-hexyl sulphenamide (*first revision*)
7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients, Part 1
7497 : 1985	High abrasion furnace (HAF) carbon black (*first revision*)
8851 : 1994	Sulphur for rubber industry (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be dark brown or purple in colour and shall be in flake/pastilles/beads or any other physical form as agreed to between the supplier and the manufacturer.

NOTE — Due to its low melting point, the material is likely to get compacted when ambient temperatures exceed 35°C during storage or transit.

3.2 The material shall comply with the requirements given in Table 1 when tested according to methods prescribed in col 4 of the table.

3.3 Compounding Performance Requirement

The material when compounded and tested as given in Annex C, shall have its properties comparable within the limits prescribed in **C-4** with those of the sample complying with the requirements of **3.1** and **3.2** and approved previously by the purchaser.

Table 1 Requirements for N- (1, 3-Dimethylbutyl)-N′-Phenyl Paraphenylenediamine (6 PPD)

(*Clause* 3.2)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to (4)
i)	Assay : N-(1, 3-Dimethylbutyl)-N′-phenyl paraphenylenediamine content, percent by mass, *Min*	94·00	Annex A
ii)	Melting point (final), °C, *Min*	45·0	A-3 of IS 6918 : 1972
iii)	Heat loss at 70°C- 3 hours, percent by mass, *Max*	0·5	Annex B
iv)	Ash content, percent by mass, *Max*	0·10	10 of IS 7086 (Part 1) : 1973
v)	Copper content, percent by mass, *Max*	0·002	12 of IS 7086 (Part 1) : 1973
vi)	Manganese content, percent by mass, *Max*	0·002	11 of IS 7086 (Part 1) : 1973

4 PACKING AND MARKING

4.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier or in 20 kg or 50 kg packing.

4.2 Marking

The packages shall be securely closed and legibly marked with the following information:

a) Name of the material,

b) Indication of the source of manufacture,

c) Net mass of the material,

d) Lot and batch number, and

e) Month and year of the manufacture.

4.2.1 The package may also be marked with the Standard Mark.

4.2.2 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The representative sample of the material shall be drawn as prescribed in IS 7086 (Part 1) : 1973.

5.2 Tests for all the characteristics shall be conducted on a composite sample.

5.3 Criteria for Conformity

The lot shall be declared as conforming to this specification if the sample satisfies all the requirements for the various characteristics given in Table 1 and also meets the specified requirements given in Annex C.

6 TEST METHODS

6.1 The test shall be conducted according to the method prescribed in Table 1. Reference to the relevant annexes and Indian Standard specifications are given in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise 'pure chemicals' and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ASSAY OF N-(1, 3-DIMETHYLBUTYL)-N'-PHENYL PARAPHENYLENEDIAMINE (6 PPD) CONTENT BY GAS CHROMATOGRAPHY

A-0 OUTLINE OF THE METHOD

This method determines the purity of N-(1, 3-dimethylbutyl)-N'-phenyl paraphenylenediamine (6 PPD) by gas chromatography (GC) using thermal conductivity detector by using diethyl phthalate as an internal standard. Approximately 1 g of the test sample of N-(1, 3-dimethylbutyl)-N'-phenyl paraphenylenediamine (6 PPD) is accurately weighed and dissolved in previously accurately weighed (approx 1 g) Diethyl phthalate (internal standard) and known volume of this solution is analyzed by programme temperature gas chromatography using 1 525 mm × 3 2 mm O.D stainless steel column packed with 3 percent SE-30 on 80-100 mesh. Chromosorb G. A. W. DMCS and a thermal conductivity detector. Peak areas are determined using integrator or laboratory data system.

This method determines the purity of N-(1, 3-dimethylbutyl)-N'-phenyl phenylenediamine (6 PPD) and assumes that all the components of interest are completely eluted from the column.

A-1 EQUIPMENT

Any high quality gas chromatograph equipped with a thermal conductivity detector and suitable integrator and recorder.

A-2 TEST CONDITIONS

The GC shall be adjusted to following test conditions:

Carrier gas	Ultra high pure hydrogen gas
Flow rate	30 ml/min
Injection port temperature	300°C
Oven programmed	150°C to 310°C (rate of increase of temperature =8°C/min)
Detector temperature	350°C
Chart motor	0·5 cm/min
Other controls	As prescribed in the instrument manual

A-2.1 Column Details

Column Dimensions — 1 525 mm × 3·2 mm in dia stainless steel tube packed with 3 percent SE on 80-100 mesh chromosorb G.A.W. DMCS.

A-3 PROCEDURE

A-3.1 Calibration

Weigh 1·0 ± 0·05 g of standard sample of N-(1, 3-dimethylbutyl)-N′-phenyl paraphenylenediamine (6 PPD) and 1·0 ± 0·05 g of diethylphthalate (GLC grade Internal Standard) accurately to nearest 0·000 1 g into a 25-ml serum bottle. Stopper, seal and shake the bottle to ensure complete dissolution. If necessary warm the contents on water bath. Inject 0·5 microlitre of this solution into the injection port and start the temperature programme and integrator recorder system to trace the peaks of components. It is recommended that this procedure be repeated till minimum five test results are obtained within 0·25 percent accuracy. Take the average of these five readings and calculate the area response factor of the material as follows:

Area response factor (ARF)

$$= \frac{\text{Mass of standard material}}{\text{Area of standard material}} \times \frac{\text{Area of diethyl phthalate}}{\text{Mass of diethyl phthalate}}$$

A-3.2 Sample Testing for Assay Determination

The test procedure adopted is similar to that described in **A-3.1** except for following change:

—the standard sample of N-(1, 3-dimethylbutyl)-N′-phenyl paraphenylenediamine (6 PPD) is replaced by the test sample (Ketone free), representing the lot of material to be tested.

A-3.2.1 The assay of the sample is calculated as follows:

$$\text{Assay of test sample, percent by mass} = \frac{\text{Mass of diethyl phthalate}}{\text{Area of diethyl phthalate}} \times \frac{\text{Area of test sample} \times \text{ARF} \times 100}{\text{mass of test sample}}$$

NOTE — All test results are reported to the nearest 0·01 percent.

Column with 3 percent SE-30 on 80-100 mesh chromosorb G.A.W. DMCS are to be conditioned before use at 300°C for 24 hours.

ANNEX B

[*Table* 1, *Item* (iii)]

DETERMINATION OF HEAT LOSS AT 70°C

B-1 PROCEDURE

Weigh accurately about 5·0 g of the material in a previously weighed flat bottom dish about 80 mm in diameter. Keep the dish with the material in an oven maintained at 70°C ± 1°C for 3 h. At the end of this period transfer the dish to a desiccator and cool. Remove the dish and weigh. Repeat the heating and weighing till the mass is constant. Express the loss in mass as the percentage of the material taken for the test.

ANNEX C

(*Clause* 3.3)

METHOD FOR COMPOUNDING AND TESTING N-(1, 3-DIMETHYLBUTYL)-N′-PHENYL PARAPHENYLENEDIAMINE (6 PPD)

C-1 TEST COMPOUND

For guidance, the test compound with the following composition may be used for testing performance properties of N-(1, 3-dimethylbutyl)-N′-phenyl paraphenylenediamine (6 PPD) in rubber compound:

	Parts by Mass
Natural rubber grade IS NR 5 (*see* IS 4588 : 1986)	100
Zinc oxide (*see* IS 3399 : 1993)	3·5
Stearic acid (*see* IS 1675 : 1971)	3·0
Rubber process oil (Naphthenic type)	3·5
HAF carbon black (*see* IS 7497 : 1985)	50·5
Sulphur (*see* IS 8851 : 1994)	2·5
Benzothiazyl-2-cyclohexyl sulphenamide (*see* IS 7069 : 1986)	0·5
N-(1, 3-dimethylbutyl)-N′-phenyl paraphenylene-diamine (6 PPD)	1·0

C-2 COMPOUND

Follow the procedure prescribed in NR : 9 of IS 3660 (Part 51) : 1972.

C-3 TESTS

C-3.1 The tests given below are recommended for each test sample. The approved sample shall also be tested in the same compound side by side.

C-3.2 Tensile strength, modulus of 300 percent elongation and elongation at break at different cures at 141°C stepwise, from below to above the expected optimum cure, shall be determined to ascertain the optimum cure time.

Tensile properties (that is, tensile strength and elongation at break) of the vulcanizates cured at the ascertained optimum cure time are determined both before and after heat ageing at 70°C for 2, 4 and 6 days.

C-4 RESULTS

The tensile strength and elongation at break, both of original sample and the aged samples shall be within ± 10 percent of the values obtained with the approved sample tested at the same time.

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